

New Compact Discrete Dynode Multipliers Integrated into the Thermo Scientific TRITON Variable Multicollector Array

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Introduction

The ^{187}Re to ^{187}Os system gives information on the formation of planetary interiors, because both elements have siderophile characters. For the Re-Os isotope systematics, precise measurements of the $^{187}\text{Os}/^{188}\text{Os}$ isotope ratio are the major target. Analytical methods need to be sensitive and accurate, because the abundances of Re and Os are very low in the ng/g range or even in the (pg/g range) and variations of the $^{187}\text{Os}/^{188}\text{Os}$ in silicate rocks are in the range of < 1%. For Negative Thermal Ionization Mass Spectrometry (N-TIMS) ionization efficiencies in the range of 10-20% can be achieved, thus making Thermal Ionization Mass Spectrometry (TIMS) the ideal analytical technology for high precision Os isotope ratio measurements of extremely small samples.

The small sample amounts require the use of ion counting detectors, where every single ion is detected and counted. In a Multi Ion Counting (MIC) setup, an array of ion counters is placed in the focal plane of the mass analyzer to allow simultaneous detection of all ion beams of interest.

Compared to a single collector approach, where all ion beams are measured sequentially on the same ion counter, the MIC approach offers ultimate detection efficiency, because of the parallel detection of the isotopes.

In this study we evaluate the performance of an improved Multi Ion Counting (MIC) setup for Os isotope ratio measurements using an array of new Compact Discrete Dynode (CDD) multipliers installed into the Thermo Scientific TRITON TIMS. Previously flat channeltron type detectors have been used for this application. However, the new CDD multipliers give improved stability and improved linearity over a larger dynamic range. The attainable precision and accuracy will be discussed and a comparison to single collector peak jumping data will be given.

Key Words

- TRITON
- TRITON Plus
- NEPTUNE Plus
- New Ion Counters
- Compact Discrete Dynode (CDD)

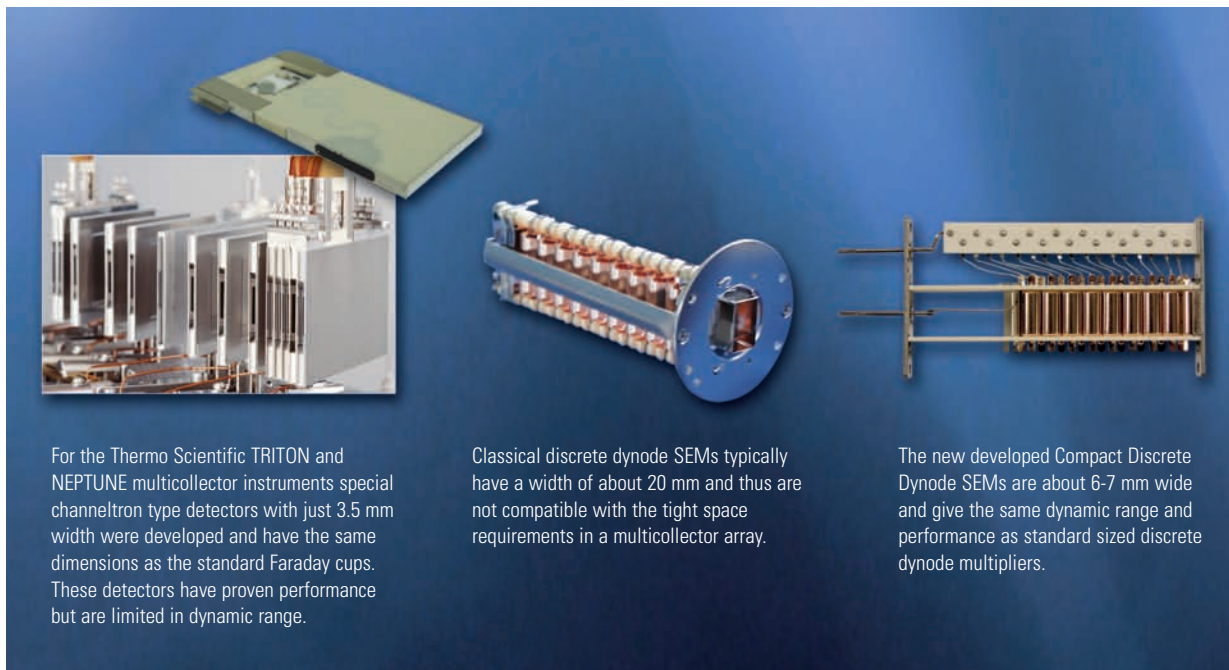


Figure 1: Three different ion counter types integrated into the Thermo Scientific TRITON multicollector.

Experimental Setup

The new Compact Discrete Dynode multipliers are about 7 mm wide and thus have about twice the width of the standard TRITON Faraday cups. Nevertheless, these new CDD multipliers can be setup to measure OsO₃ isotopes at one amu spacing. This is due to the asymmetrical input aperture of the CDD multipliers, see Figure 2.

Cup Configuration

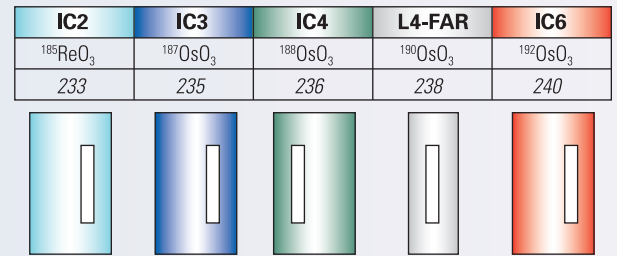


Figure 2: Cup Configuration for OsO₃ using CDDs.

Results

Dark Noise and Yield

	IC2	IC3	IC4	IC6
Darknoise	0.171 cpm	0.19 cpm	0.171 cpm	0.247 cpm
Yield	98.3%	98.5%	99.5%	97.3%

Table 1: Darknoise & counting efficiency of Compact Discrete Dynode SEMs.

Darknoise Determination IC 3
Duration: 60.00 [Min] Integration Time: 16.78 [s]
SEM/IC Operating-Voltage: 1575.0 [V]

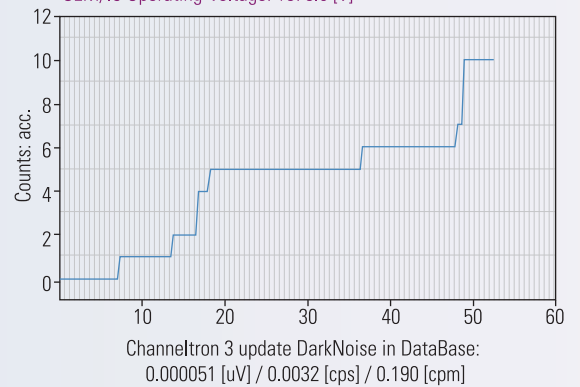


Figure 5: Darknoise determination. Total measurement time: 60 minutes.

Results

Linearity

¹⁹² OsO ₃ / ¹⁸⁸ OsO ₃	¹⁸⁷ OsO ₃ / ¹⁸⁸ OsO ₃	
IC6 intensity	IC6/IC4	IC3/IC4
50 kcps	3.05659	0.10750
100 kcps	3.05604	0.10747
200 kcps	3.05678	0.10742
400 kcps	3.05672	0.10745
800 kcps	3.05645	0.10743
RSD	0.03%	0.03%
All ion counter dead times set to 23 ns.		

Table 2: OsO₃ Isotope ratios measured at different intensities.

Cross Calibration

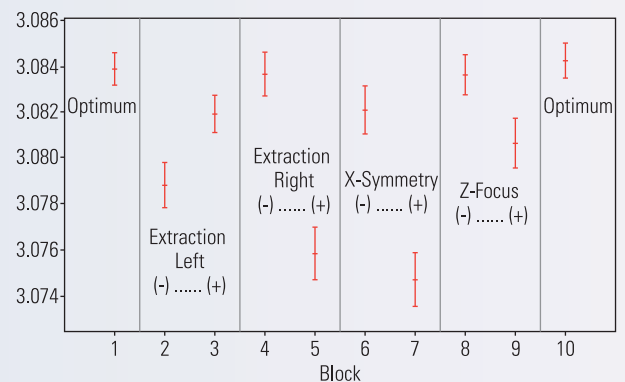


Figure 8: Robustness of cross calibration factors against source detuning: Ion source was intentionally detuned to 50% sensitivity.

Results

Plateau Calibration

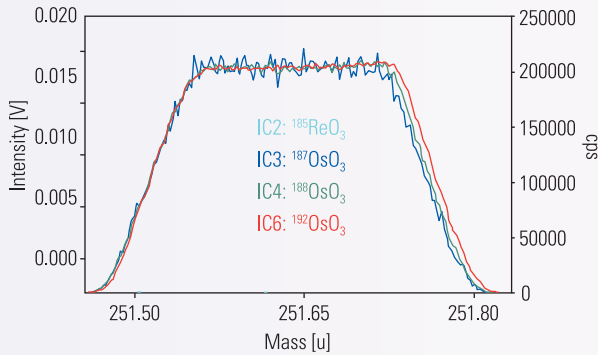


Figure 3: OsO_3 Peak overlap using Compact Discrete Dynode SEMs. Mass scale is related to the centre of the multicollector system. Counter intensities are normalized to IC6 (200 kcps).

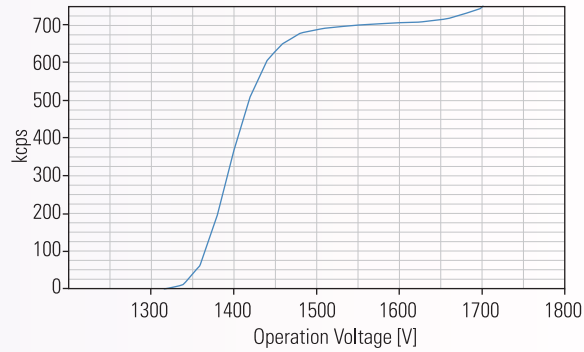


Figure 4: Typical plateau curve of Compact Discrete Dynode SEMs.

Stability

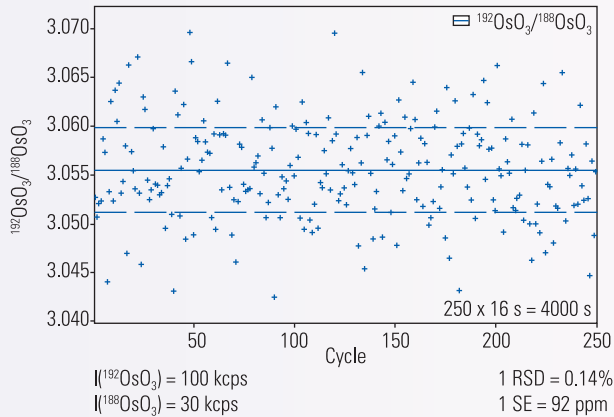


Figure 6: Relative stability at 100 kcps for Compact Discrete Dynode SEMs.

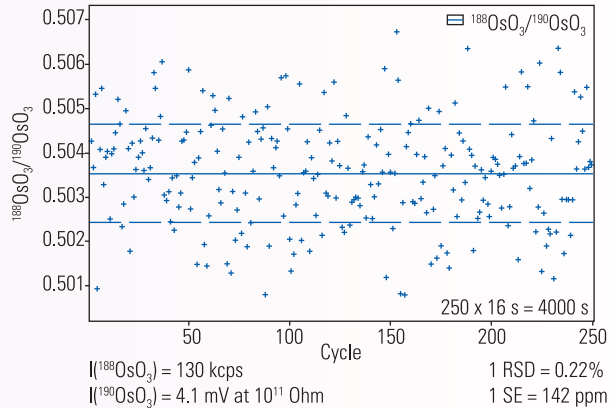


Figure 7: Absolute stability: Compact Discrete Dynode SEM versus Faraday cup.

As a robustness test the ion source lenses have been intentionally detuned (50% sensitivity loss) to investigate any influence of source tuning parameter on cross calibration factors. The measured isotope ratio never changed by more than 0.3%, regardless of any source detuning. In reality one would always aim to work with highest sensitivity tuning and thus source tuning effects will be much reduced. Therefore, these tests can be regarded as worst case scenarios.

	IC2	IC3	IC4	L4-FAR	IC6	Integration time
Static line 1	$^{185}\text{ReO}_3$	$^{187}\text{OsO}_3$	$^{188}\text{OsO}_3$	$^{189}\text{OsO}_3$	$^{192}\text{OsO}_3$	16 s
Jump 1			$^{192}\text{OsO}_3$			1 s
Jump 2		$^{192}\text{OsO}_3$				1 s
Jump 3	$^{192}\text{OsO}_3$					1 s

Table 3: „In-run“ cross calibration.

Conclusions and Summary

Advantages

- Collection of all isotopes at the same time
- Works well in case there is one major isotope
- Corrects for „in-run“ calibration changes

Disadvantage

- Precision tied to a single collector measurement for TIMS

Sample/Standard Calibration

Here the cross calibration factors are deduced from the measurement of a known standard prior to the measurement of the sample. Since the cross calibration factors are rather robust against different source tuning conditions, this procedure should work well to better than 0.2%.

Performance Indications of the new CDD Multipliers

- Dynamic range: 1 cps to 1.400.000 cps
- Linearity: < 0.2% over full dynamic range
- Dark noise: < 10 cpm (0.2 cpm typical)
- Stability: < 0.2% drift per hour at 100 kcps
- Peak flatness: < 0.2% over 150 ppm in mass

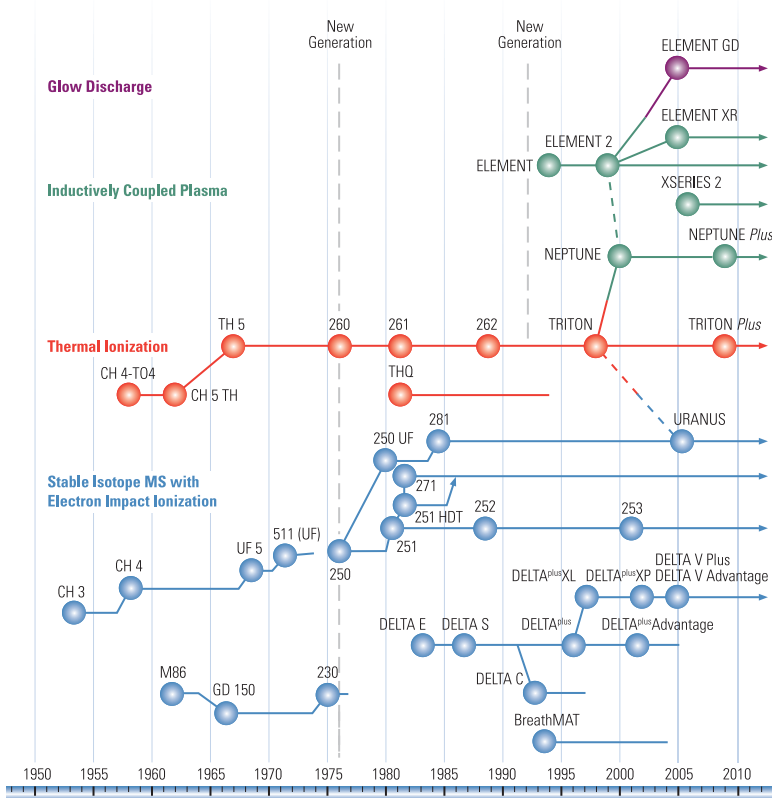
Cross Calibration

- Cross calibration is robust against source tuning
- “In-run” cross calibration works, but is finally limited to single collector calibration
- *Sample/Standard* calibration is very promising: stabilities of the ion counters are good enough to achieve sub permil precisions.

Faraday Cups

- Faraday cups use electronic cross calibration and are the work horse as well as the solid reference for critical analysis results.
- High gain amplifiers (> 10¹¹ Ohm) can bridge the gap between ion counting and Faraday cup measurements.

History of Inorganic Product Lines



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